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(54) **MOLTEN-SALT-BASED GROWTH OF GROUP III NITRIDES**

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117/108; 117/952

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See application file for complete search history.

(56) **References Cited**

U.S. PATENT DOCUMENTS

- 6,127,280 A * 10/2000 Stutz 438/746
6,139,629 A * 10/2000 Kisielowski et al. 117/105
6,218,280 B1 * 4/2001 Kryliouk et al. 438/607
6,265,322 B1 * 7/2001 Anselm et al. 438/745
6,379,472 B1 * 4/2002 Kisielowski et al. 148/33.1
6,884,740 B2 * 4/2005 Hu et al. 438/708

2003/0045120 A1 * 3/2003 Hu et al. 438/745
2006/0048701 A1 * 3/2006 Feigelson et al. 117/89
2007/0248526 A1 * 10/2007 Spencer et al. 423/412

OTHER PUBLICATIONS

Yujiang Song et al, "Preparation and Characterization of bulk GaN Crystals" Journal of Crystal Growth, 206 (2004), pp. 327-330.

Toru H. Okabe et al, "Electrochemical Properties of Li₃N Dissolved in Molten LiCl at 900 K", Journal of The Electrochemical Society, 148 (5) (2001) pp. E219-E226.

T. Ogawa et al, "Dissolution and Formation of Nuclear Materials in Molten Media", Pure and Applied Chemistry vol. 73, No. 5, 2001, pp. 799-806.

Izabella Grzegory, "High Pressure Growth of Bulk GaN from Solutions in Gallium", Journal of Physics Condensed Matter, vol. 13, 2001, pp. 6875-6892.

A.D. Franklin, "Electrochemical Growth of Crystals from Electrolyte Solutions," Journal of Crystal Growth, 34 (1976), pp. 245-247.

Takuya Goto et al, "Electrochemical Reduction of Nitrogen Gas in a Molten Chloride System", Electrochimica Acta, vol. 43, Nos. 21-22, pp. 3379-3384, 1998.

(Continued)

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ABSTRACT

A method for growing Group III nitride materials using a molten halide salt as a solvent to solubilize the Group-III ions and nitride ions that react to form the Group III nitride material. The concentration of at least one of the nitride ion or Group III cation is determined by electrochemical generation of the ions.

32 Claims, 5 Drawing Sheets

